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ऊनी और रेशमी कपड़ो की धुलाई के लिए संश्लेषिक अपमार्जक — विशिष्टि

(दूसरा पुनरीक्षण)

Synthetic Detergents for Washing Woollen and Silk Fabrics — Specification

(Second Revision)

ICS 71.100.40

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Soaps and other surface active agents Sectional Committee, CHD 25

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps and other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Woollen, silken and other delicate fabrics, unlike cotton fabrics, are sensitive to alkalis. Conventionally, therefore, neutral soap without builders and practically free from caustic alkali has been used for washing woollens, silken and synthetic fabrics in households and laundries. With the introduction of synthetic detergent based products, the position has changed.

This Indian Standard was originally published in 1980 and subsequently revised on 1994. In first revision, type 1 namely, powder had been deleted as it was not being manufactured or marketed in the country. With a view to ensure safety from skin irritation and skin sensitization hazards to consumers using this product and to ensure that this is also not injurious to the fabrics washed, a requirement for safety of the product on use had also been introduced in first revision.

Presence of phosphorous in excess in water body is known to cause eutrophication leading to algal growth, thereby posing risks to aquatic life. Phosphorous based compounds (for example, phosphates, phosphonates etc.) used in detergent products can contribute to the above. Therefore, in pursuit of minimizing the environmental impact, the committee responsible for formulation of this standard has decided to revise it again. Hence, besides amalgamation of all amendments, the maximum limit of phosphate has been restricted as ‘nil’ in this revision as liquid detergents usually contain mild cleansers and surfactants like sodium laureth sulphate, non-ionics etc.There is no need for adding phosphate based builders in the formula. Performance requirements like detergency and ash built up had also been incorporated in this revision. Further, the use of Zeolite, which is a non-phosphate based builder and use of co-builders enzymes (bio-based actives) for detergent has been recommended as an alternative to phosphate based builder.Tri-sodium citrate has also been incorporated in list of conventional builders and additives since it has the potential to replace phosphates and is more environment friendly than the phosphates.

It is necessary that the raw materials used in the formulation of detergents are such that in the concentration in which they will be present in the finished product, after interaction between them are free from any harmful effects. For determining the suitability of a new formulation or of a new raw material used in formulations for skin safety, necessary tests as prescribed in IS 11601 : 2002 Methods of safety evaluation of synthetic detergents — Tests for skin irritation and sensitization potential of synthetic detergents need to be followed.

A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of Ministry of Environment and Forest & Climate Change (MoEF & CC). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 2016 as per the Resolution No 71 dated 20 February 1991 published in the Gazette of the Government of India. For a product to be eligible for ECO Mark, it shall carry the standard mark of BIS for quality besides meeting additional optional environment friendly (EF) requirements.

This standard was formulated by CHD 25 technical committee. The list of experts who made significant contribution to the revision of this standard is given at Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SYNTHETIC DETERGENTS FOR WASHING WOOLLEN AND SILK FABRICS — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for synthetic detergents for washing woollen and silk fabrics.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards indicated below:

IS No	*Title*
264 : 2005	Specification for nitric acid (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
4707 (Part 1) : 2017	Classification for cosmetic raw materials and adjuncts Part 1 Colourants (*third revision*)
4707 (Part 2) : 2017	Classification for cosmetic raw materials and adjuncts: Part 2 List of raw materials generally not recognized as safe for use in cosmetics (*fourth revision*)
4955 : 2001	Household Laundry detergent powders (*fourth revision*)
4956 : 2002	Synthetic detergent for industrial purposes (*second revision*)
5785 (Part 4) : 1976	Methods of performance tests for surface active agents: Part 4 Relative detergency (*first revision*)
7597 : 2001	Surface activity agents — Glossary of terms (*first revision*)
8401 : 1994	Alkyl benzene sulphonic acid (acid slurry) — Specification (*first revision*)
11601 : 2002	Methods of safety evaluation of synthetic detergents — Tests for skin irritation and sensitization potential of synthetic detergents (*first revision*)
12795 : 1989	Linear alkyl benzene — Specification
13933 : 1995	Method of test for biodegradability of surface active agents (modified Sturm test)
15267 : 2003	Zeolite detergent grade — Specification

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7597 shall apply.

4 REQUIREMENTS

4.1 Composition

4.1.1 The active ingredient used in the formulation of the material shall be one or more of the surface active chemicals namely, alkyl benzene sulphonate, secondary alcohol sulphate, fatty alcohol sulphate, fatty alcohol ethoxylate, salts of sulphated fatty alcohol ethoxylate, sodium alpha olefin sulphonate, and alkylphenol ethoxylate. The formulation may contain one or more of the builders and additives given in Annex A, or any other builders or additives keeping in view the end use of the product.

4.1.2 The material shall not contain any plastic microbeads or other synthetic abrasive material.

4.2 Description

The material shall be clear liquid and free from any unpleasant odour, and shall possess good lathering and cleaning properties.

4.3 Alkyl Benzene Sulphonic Acid

Alkyl benzene sulphonic acid from which alkyl benzene sulphonate is prepared shall conform to IS 8401 for which linear alkyl benzene conforming to IS 12795 shall be the starting material.

4.4 Safety Evaluation

4.4.1 The material may contain permitted colour as given in IS 4707 (Part 1). The material shall not contain any ingredient above the limit as given in IS 4707 (Part 2).

4.4.2 The material shall pass the test for skin irritant and sensitization potential when evaluated as per the method prescribed in IS 11601.

4.5 The material shall also comply with the requirements given in Table 1.

4.6 Additional Requirements for ECO Mark

4.6.1 *General Requirements*

4.6.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under **4.1** to **4.5** except that for phosphate content which shall be substituted with alternate environment friendly builder(s) to maintain similar detergency when tested according to IS 5785 (Part 4).

4.6.1.2 The manufacturers shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the *Water (Prevention and Control of Pollution) Act*, 1974 and *Air (Prevention and Control of Pollution) Act*, 1981 along with the authorization, if required under *Environment (Protection) Act*, 1986, while applying for ECO Mark.

4.6.2 *Specific Requirements*

4.6.2.1 The material shall not contain any phosphate when tested as per the method prescribed in Annex D of IS 4955. Any other substitute used shall be environmental friendly but should be in sufficient quantity to ensure similar performance of the product as compared to that of phosphates.

4.6.2.2 The material shall pass the test for skin irritant and sensitization potential when evaluated as per the method prescribed in IS 11601.

4.6.2.3 The surfactants used in the manufacture of synthetic detergent for washing woollen and silk fabrics shall be readily biodegradable when tested by modified Sturm test as prescribed in IS 13933.

5 PACKING AND MARKING

5. 1 Packing

5.1.1 The material shall be suitably packed as agreed between interested parties. The packing shall be capable to prevent any moisture absorption.

5.1.2 For ECO Mark, the product shall be packed in such packages which are made from recyclable/reusable or biodegradable materials and declared by the manufacturer and may be accompanied with detailed instructions for proper use.

5. 2 Marking

Each package shall be securely closed and marked with the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material, when packed;
d) Batch number or lot number in code or otherwise;
e) Month and year of manufacture;
f) Shelf life of the product;
g) **A CAUTIONARY NOTICE**: Detergent solutions can be skin irritants. Avoid prolonged contact. Rinse garments and hands thoroughly; and
h) Critical ingredients mentioning the actual compound in descending order up to a limit of 0.5 percent by mass:
    1) Active ingredients;
    2) Builders used;
    3) Fillers;
    4) Soda ask
    5) Enzymes, if added; and
    6) Bleach and bleach activators, if added.

In addition to above, product may also be marked with details as per the requirement of legal metrology regulations and any other applicable statute.

5.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5.4 The following information shall be marked on the label for ECO Mark:

a) The criteria for which the product has been labelled as ECO Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in Annex H of IS 4956.

7 TESTS

7.1 Tests shall be conducted as prescribed in IS 4955 and IS 4956 and Annex B of this standard, reference to which has been given in co1 **4**, **5** and **6** of Table 1.

7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results, of analysis.

Table 1 Requirements for Synthetic Detergents for Washing Woollen and Silk Fabrics

(*Clauses* 4.5 and 7.1)

Sl No.	**Characteristics**	**Requirements**	**Method of test, Ref to**		
			Annex of this Standard	Annex in IS 4955	Annex in IS 4956
(1)	(2)	(3)	(4)	(5)	(6)
i)	Active ingredient percent by mass, *Min* (*see* note 1)	15	B	–	–
ii)	Total phosphates content, expressed as STPP, calculated from phosphorus pentoxide, percent by mass, *Max* (*See* note 2)	nil	–	D	–
iii)	pH of 1 percent solution (*m*/*v*) at 27°C	7 to 8.5	–	–	F
iv)	Matter insoluble in water, percent by mass, *Max*	Nil	–	–	G
v)	Clear point, °C, *Max*	10	–	–	E
vi)	Percent detergency *Min*	65		G	
vii)	Ash built up, percent *Max*	1		H	

NOTES:

1) In case non-ionics and soaps are absent, the active ingredients shall be determined by the method given in Annex B of IS 4955.

2) Use of natural builder such as Zeolite (*see* IS 15267) is recommended.

ANNEX A

(*Clause* 4.1)

LIST OF CONVENTIONAL BUILDERS AND ADDITIVES

1 Crystalline sodium aluminosilicate (zeolite)
2 Polyvinyl pyrrolidone
3 Trisodium Citrate
4 Sodium carboxymethyl cellulose
5 Sodium silicate
6 Hydrotropes
7 Colours
8 Foam boosters
9 Organic solvents such as isopropanol and ethyl alcohol
10 Perfume
11 Preservatives
12 Chelating agents (sequestering agents)
13 Optical brighteners
14 Enzymes

ANNEX B

[*Clause* 7.1, *and Table* 1, *Sl No.* (i)]

DETERMINATION OF ACTIVE INGREDIENTS

B-1 GENERAL

Active matter is first separated from inorganic salts, non-detergent organic matter and alkylamide. It is then neutralized to phenolphthalein, evaporated to dryness, extracted with ethyl alcohol, dried and weighed. Finally, the weighed extract is corrected for the presence of sodium chloride and alkali carbonates.

B-2 APPARATUS

B-2.1 Beakers, 150 ml and 1 000 ml capacity.

B-2.2 Buchner Flask, 500 ml capacity, fitted with a sintered glass filter funnel (porosity 4).

B-2.3 Evaporating Dish

B-2.4 Separating Funnels, 1 000 ml capacity.

B-2.5 Steam-Bath

B-2.6 Wide-Mouthed Flat-Bottomed Flask, 200 ml capacity.

B-2.7 Air-Oven, preferably electrically heated, along with temperature control and display device.

B-3 REAGENTS

B-3.1 Ethyl Alcohol, 30 percent, 90 percent, 96 percent and absolute (by volume).

B-3.2 Diethyl Ether

B-3.3 Acetone

B-3.4 Standard Sulphuric Acid, 0.1 N.

B-3.5 Standard Silver Nitrate Solution, 0.1 N.

B-3.6 Phenolphthalein Indicator, 1 percent solution in 95 percent (by volume) ethyl alcohol.

B-3.7 Methyl Orange Indicator, 0.1 percent (m/v).

B-3.8 Nitric Acid, concentrated, relative density 1.42 (conforming to IS 264).

B-3.9 Nitrobenzene

B-3.10 Standard Ammonium Thiocyanate, 0.1 N

B-3.11 Ferric Ammonium Sulphate Indicator, saturated solution.

B-4 PROCEDURE

B-4.1 Removal of Inorganic Salts

Weigh accurately about 6 g of material containing about 15 percent active matter and if product contains less or more of active matter content, weigh the sample proportionally or pro-rata. Digest with 60 ml of ethyl alcohol by heating on a steam-bath for about 2 min. Stir and break up any hard lump with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel no.4 fitted to a Buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30 ml portions of boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and, finally wash the residue several times with hot ethyl alcohol to remove all the alcohol soluble. Collect the filtering and washing of the residue and evaporate the combined filtrate to a small bulk in an evaporating dish.

B-4.2 Removal of Non-detergent Organic Matter and Alkylolamide

B-4.2.1 Dilute the evaporated filtrate (*see* **B-4.1**) with 50 ml of 90 percent ethyl alcohol, and transfer to a separating funnel. Rinse the evaporating dish once with 50 ml of 90 percent ethyl alcohol and then four times with 50 ml portions of water. Add each wash in turn to the separating funnel. Add 150 ml of diethyl ether, swirl gently to ensure adequate mixing, and allow the two phases to separate. Run off the aqueous alcoholic layer into a second separating funnel, and extract twice with 75 ml portions of diethyl ether. Transfer the aqueous alcoholic phase into a beaker, and combine the three ether extracts.

B-4.2.2 Take the combined ether extracts in a clean separating funnel. Wash three times with successive 50 ml portions of 30 percent ethyl alcohol and then with successive 50 ml portions of water until the ether phase is free from alcohol, usually 7 to 10 water washes are necessary.

B-4.2.3 Combine the washings and rinsing from **B-4.2.2** with the aqueous alcoholic phase obtained in **B-4.2.1.**

B-4.3 Determination of Active Matter

B-4.3.1 Transfer the combined aqueous alcoholic phase and washings (**B-4.2.3)** to a porcelain dish. Neutralize to phenolphthalein and evaporate on a steam-bath until the volume is reduced to about 25 ml. Add an equal volume of absolute alcohol and evaporate to dryness. The solution shall remain just pink to phenolphthalein throughout evaporation.

B-4.3.2 To ensure that the residue is completely anhydrous, add 30 ml of absolute alcohol and again evaporate to dryness. Extract the residue with 30 ml of hot 96 percent ethyl alcohol, stirring and breaking up the solid matter in the dish with a glass rod. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel no. 4fitted to a Buchner flask to which suction is applied. Extract the residue in the dish with six further consecutive 30 ml portions of hot 96 percent ethyl alcohol. Pass each extract in turn through the sintered glass filter. Finally, wash the residue in the sintered glass filter three times with about 20 ml of hot 96 percent ethyl alcohol from the jet of a wash bottle.

B-4.3.3 Transfer the filtrate and washings from the Buchner flask to a tared wide-mouthed flat- bottomed flask, evaporate nearly to dryness on a water-bath, and drive off the remaining solvent by directing a gentle stream of dry-air into the flask whilst continuously rotating the later on the hot water-bath. A thin film of active matter, easy to dry, is thereby obtained. Add 10 ml of acetone, evaporate and remove the last traces of solvent as described above, cool in a desiccator and weigh.

B-4.3.4 Heat the flask for not more than 5 min in an air-oven at a temperature of 100±1°C, gently blow out with a current of air, cool and reweigh. Repeat this drying process until the difference between two successive weighings does not exceed 3 mg. Record this mass as M_1.

B-4.3.5 The extract obtained in **B-4.3.4** contains the active matter, some sodium chloride and possibly a trace of alkali carbonates which may have passed through the filter in the presence of the detergent.

B-4.4 Determination of Alkali Carbonates

B-4.4.1 Dissolve the extract (*see***B-4.3.4**) in cold distilled water, add a few drops of methyl orange indicator and titrate with standard sulphuric acid to the methyl orange end point.

B-4.4.2 *Calculation*

Mass, in g, of sodium carbonate, $M_2 = 0.053\ VN$

Where,

V = volume of standard sulphuric acid solution used, in ml; and

N = normality of the standard sulphuric acid solution.

B-4.4.3 Preserve the solution for the estimation of chlorides.

B-4.5 Determination of Chlorides

B-4.5.1 To the solution remaining after the estimation of alkali carbonates (*see* **B-4.4.3**), add 2 ml of concentrated nitric acid and 20 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene and shake vigorously. Titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate as indicator.

B-4.5.2 *Calculation*

Mass in g of sodium chloride, $M_3 = 0.058\,5\ (20A - BC)$

where,

A = normality of the standard silver nitrate solution;

B = volume, in ml, of standard ammonium thiocyanate solution used; and

C = normality of the standard ammonium thiocyanate solution.

B-4.6 Determination of Active Matter

$$\text{Active matter, percent by mass} = \frac{100 \times (M_1 - M_2 - M_3)}{M}$$

Where,

M_1 = mass, in g, of the alcoholic extract (*see* **B-4.3.4**);

M_2 = mass, in g, of sodium carbonate (*see* **B-4.4.2**);

M_3 = mass, in g, of sodium chloride (*see* **B-4.5.2**); and

M = mass, in g, of the material taken for the test.

ANNEX C

(*Foreword*)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

Organization	*Representative(s)*
Harcourt Butler Technical University, Kanpur	Prof Rakesh Trivedi (***Chairman***)
Drugs Controller General of India, New Delhi	Shri Sudipta Dey
Fena (P) Ltd, New Delhi	Shri Dalip Jolly Shri Benny G. Jacob (*Alternate*)
Hindustan Unilever Ltd, Mumbai	Dr A. Sivakumar Ms Vrinda Rajwade (*Alternate*)
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Reckitt Benckiser India Ltd, Gurgaon	Shri Brij Mohan Singh Dr Pooja Sharma (*Alternate*)
Directorate General, BIS	Shri U. K. Das, Scientist 'E' and Head (CHD) [Representing Director General (*Ex-officio*)]

Member Secretary

Shrishti Dixit
Scientist 'C' (CHD), BIS

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